Validation of Metals for the Contract Laboratory Program (CLP) based on SOW ILMO5.3 (SOP Revision 13)



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Standard Operating Procedure
USEPA Region 2
Evaluation of Metals Data for the Contract Laboratory Program
Data Assessment and Contract Compliance Review

SOP: HW-2 Revision 13

Sept. 2006

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I.0 Scope

- I.I This Standard Operating Procedure (SOP) applies to the evaluation of Routine Analytical Services (RAS) inorganic data generated in accordance with the EPA Contract Laboratory Program (CLP) protocols.
- 1.2 This Region 2 inorganic data validation SOP is used to determine the usability of analytical data generated from water and soil/sediment samples collected from Superfund sites in EPA Region 2.
- 1.3 Data should be generated and validated in accordance with the site specific Project Quality Objectives (PQOs) developed prior to the sample collection event. This SOP can be customized to validate the data according to the site specific PQOs. If the site specific DQOs are not available, this SOP must be used in its entirety.
- 1.4 This SOP is based, for the most part, upon analytical and quality assurance requirements specified in the Statement of Work SOW-ILM05.3, as well as in the final (October 2004) of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. The SOP Checklist, Appendix A.1, provides guidance in conducting the data validation. The result of the use of this SOP is a **Total Review** of the data: **Technical plus Contract Compliance Review**.

2.0 **Contract Compliance Review**

This type of review is the first step in data validation which is carried out to ensure that the CLP laboratory has analyzed the environmental samples in accordance with the Statement of Work (SOW), and provided a data package which is both complete and compliant. This means that laboratory's procedures were performed exactly as specified in the CLP Statement of Works (SOW) and the data package contains all the deliverables including the information required under the contract.

2.1 Completeness

The data validator must check the entire data package to ensure that all deliverables required under the CLP contract are present and legible. In addition, copies of the Contract Compliance Screening (CCS) report, re-submittal from the laboratory, and Regional documentation should also be present in the data package. In Region 2, the data package completeness check is currently performed by the Regional Sample Control Coordinator (RSCC)for each Sample Delivery Group (SDG). The data package is not released to the data validator until all the required deliverables are received from the laboratory.

2.2 **Compliance**

The data validator must check to ensure that all steps from sample receipt through sample preparation, analysis, data calculation and reporting are documented, and the information/data required under the contract is present in the appropriate reporting Forms and laboratory logs.

2.3 Contract Compliance Screening (CCS)

This screening step essentially checks the data package for the Completeness and Compliance requirements, and is performed by the Sample Management Office (SMO) currently operated by Computer Sciences Corporation (CSC), an EPA contractor. The CCS Report outlines the incomplete and non-compliant items as "Defects" in the data package, and is sent to the laboratory which is required to

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provide additional or missing information/data required under the contract. The CCS Report for each SDG is transmitted electronically by the SMO to the Regional office. The CCS Report is intended to aid the data validator in locating any problems, both corrected and uncorrected. The incorrect original deliverable(s)of the data package must be replaced by the re-submittal(s)received from the laboratory in response to the CCS Report. The data validation should, however, be carried out even if the CCS Report is not available.

Web-based CCS is available for CLP laboratories to check their data prior to its delivery to EPA.

3.0 Technical Review

Technical review of the RAS data is carried out on the complete and compliant data to ensure its validity (i.e., data is of known quality and scientifically valid) and usability (i.e., data set is sufficiently complete and of sufficient quality to support a decision or an action described in the specific objectives of a data collection activity). The technical review process provides information on analytical limitations of data, if any, based on specific Quality Assurance/Quality Control (QA/QC) criteria. This is accomplished by performing an in-depth review of both the field deliverables which document the field sampling activities, and the laboratory analytical data deliverables which document the laboratory activities carried out to generate the reported data. Essentially, the validator shall first ensure that the data package is complete and compliant. The validator shall then evaluate data/information on all these deliverables (Final data sheets, Forms for QC analyses Chain-of-Custody/Traffic Report Forms, raw data, etc.) against the QA/QC acceptance criteria specified in the SOP "Checklist" (Appendix A.1). The validator must answer each question in the "Checklist" and take an appropriate action as required under "Action" to qualify the data. As a result of the technical review, the data validator may qualify some of the data as rejected or as estimated. The data validator shall write a Data Review Narrative documenting the qualified data and the reason(s) for the qualification.

- 3.1 If the **raw data** necessary to support the reported results are not provided, the data validation must not be performed. The laboratory must be contacted to obtain missing raw data.
- 3.2 If batch quality control analyses are performed on samples other than **site specific samples**, data must not be validated or at best be considered as estimated. The data user must be notified of this action.

3.3 QA/QC Acceptance Criteria

In order that reviews be consistent among reviewers, QA/QC protocol (stated in Appendix A.I) should be strictly adhered to. If a lab provides more than one set of QC analyses or more than one particular QC analysis for an SDG, the validator shall use the worst QC analysis to evaluate the SDG data. Professional judgement should only be used in the rare instances not addressed in the "Checklist".

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3.4 **Data Validation Flags**

Three types of data validation flags (J, R & U) are used in Region 2 to qualify the data.

3.4.1 Flag "R" indicates Rejected Data

Sample results determined to be unacceptable must preferably be lined over and flagged "R" with a red pencil only on the Inorganic Analysis Data Sheets (CLP Form I's). Data rejected on the basis of an unacceptable QC analysis should be excluded from further review or consideration. Data are rejected when associated QC analysis results exceed the expanded control limits of the QC criteria. The rejected data are known to contain significant errors based on documented information. The data user **must not** use the rejected data to make environmental decisions.

3.4.2 Flag "J" indicates Estimated Data

Sample results determined to be estimated must be flagged "J" with a red pencil only on the CLP Form I's. Data are flagged (J) when a QC analysis falls outside the <u>primary</u> acceptance limits. The qualified "J" data are not excluded from further review or consideration. However, only one flag (J) is applied to a sample result even though several associated QC analyses may fail. The "J" data may be biased high or low.

3.4.3 Flg "U" indicates Non-Detects

Sample results ≥ MDL associated with a contaminated blank are flagged "U" with a red pencil only on Form I's.

4.0 Contractual Qualifiers

The CLP laboratory applies contractual qualifiers on all Form I'S and the QC Forms when QC analyses are outside the control limits. These qualifiers are not applied on the Lotus or XLS spreadsheets with the exception of U and J. The contractual qualifiers and their meanings are as follows:

- N: This qualifier indicates the lack of accuracy in the reported result, and is applied when matrix spiked sample recovery is outside the control limits.
- E: This qualifier indicates the presence of interference, and is applied when the ICP serial dilution analysis is outside the control limits.
- *: This qualifier indicates the lack of precision, and is applied to sample results on Form I's and Form VI when the Lab Duplicate analysis is outside the control limits.
- U: This is a concentration qualifier that laboratory applies to a non-detected result which is essentially less than the Method Detection Limit(MDL). A non-detected result of an analysis is indicated by the Contract Required Quantitation Limit (CRQL) of that analyze suffixed with "U".

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J: This is a concentration qualifier that the laboratory applies to a positive result below the CRQL(i.e., MDL but <CRQL).

NOTE: The laboratory qualifiers are crossed out and replaced with the appropriate data validation qualifiers (J, R or U) by the data validator.

4.0 Rounding Rule

The data reviewer must follow the standard practice to round off percent recoveries on the QC reporting forms.

5.0 **Data Review Narrative (Appendix A.2)**

The data review narrative should be written using the format of Appendix A.2. The narrative should indicate the QC analyses outside the acceptance limits and the actions taken to qualify the associated data. The narrative should be prepared on a Personal Computer or a typewriter. If hand-written, under no circumstances should a pencil be used to write the narrative. The Data Review Narrative should be written in four (4) Sections: (i)Data Case Description, (ii)Complete SDG File (CSF) Audit Section, (iii) Technical Review Section, and (iv) Contract-Problems/Non-Compliance Section.

5.1 **Data Case Description Section**

The data validator must briefly describe the data case in this Section, outlining important information such as the number of samples, their matrix, sampling date(s), analysis (TAL metals, mercury or cyanide), samples used for QC analyses, Field Blank(s), Field Duplicates, etc.

5.2 Complete SDG File (CSF) Audit Section

The data validator must perform an audit on each SDG in the data package to ensure that all SDG-specific documents (sampling, samples shipping and receiving, telephone contact logs, etc.) are present in the data case. The audit shall also discover any discrepancy in the deliverables. In Region 2, this audit is currently performed by the ESAT data validator and its findings reported under "Comments" on a CSF inventory checklist. The validator informs the CLP Project Officer (PO) of the missing or additional information/deliverable required for data validation. The PO then contacts the lab for the desired deliverable/information. The findings of the CSF audit are reported in the CSF Section of the Data Review Narrative (Appendix A.2).

5.3 **Technical Review Section**

The data validator shall report in this Section only the rejected (R) and estimated data (J) and the data rendered non-detects (U) as a result of technical review. It is imperative that the data reviewer highlights (i) QC analysis criteria applied to reject (R) or flag (J, U) the data, (ii) Samples rejected (R) or flagged (J, U), and (iii) the QC analysis out of control limits. The rest of the data that are not qualified (rejected or estimated) are not reported in this Section, and should be considered **fully useable**.

5.4 Contract-Problems/Non-Compliance Section

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All the CLP non-compliant items detected during data review must be reported in this Section.

6.0 Computer-Aided Data Review and Evaluation (CADRE)

CADRE is a computer program that performs semi-automated Quality Assurance (QA) and Quality Control (QC) checks of results from the chemical analysis of soil and water samples according to the CLP protocols. After the CADRE data qualification is complete, a Lotus 1,2,3 spreadsheet or an XLS spreadsheet with data validation qualifiers (R,J,U) is generated for each SDG. Currently, Sample Management Office (SMO) performs this task using Data Assessment Tool (DAT), a software-driven process, and forwards to the Regions the customized electronic spreadsheets (Lotus 1,2,3 or XLS spreadsheet) and QC reports via the DART (Data Assessment Rapid Transmittal) system. Manual data validation is performed in conjunction with electronic data validation which can only be done by a trained and experienced data validator. The manual data review complements CADRE's findings to complete an assessment of data quality in a shorter time than by a solely manual process. The data validator must review the XLS or Lotus 1,2,3 spreadsheet against Form I's to ensure that the same results on Form I's and the Spreadsheet are qualified with the same data validation qualifiers. The spreadsheet for each SDG is provided with the Data Review Narrative.

7.0 Performance Evaluation Sample(PES)Based Data Validation Strategy

7.1 **Scope and Summary**

This strategy offers the use of Performance Evaluation Samples (PES) in the data validation process as a means of ensuring the quality of the CLP data while significantly reducing the validation time. The single blind PES provided by EPA (or any other reputable firm) is analyzed with samples of each matrix in a Sample Delivery Group (SDG). A software program (e.g.,PEAC TOOLS, SPS Web or equivalent) is used to determine whether or not the PES results fall within the previously statistically determined acceptance limits ("Action Low" and "Action High") for the Contaminants of Concern (COC). The PES results falling within the Action Limits are considered as acceptable results and may be designated as "Passed" analytes, and results of the analytes falling outside the Action Limits are considered as unacceptable and may be designated as "Failed" analytes. In either case ("Passed" Analytes or "Failed" analytes), the associated data is validated according to the Region 2 data validation SOP HW-2 in conjunction with the latest version of the WinCadre QC reports. The following strategy (procedure) is used:

7.2 "Passed" COC

If the COC in an SDG are within statistically generated Action Limits, the data validation is conducted according

to QC analyses indicated by check marks ($\sqrt{\ }$)in the "Review COC For" column of the Table I. The SDG samples are validated using the Region 2 data validation SOP in

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conjunction with the latest version of the WinCADRE QC reports. The validation flags (J, R, U) are applied on Form I's as well on the CADRE Lotus 1,2,3 or XLS spreadsheet. Corrections, if needed, are

then made on the Lotus or XLS spreadsheet to ensure that all results on Form I's carry the same data validation and concentration flags as are on the Lotus or XLS Spreadsheet.

7.3 "Failed" COC

If the COC in an SDG are not within the statistically generated Action Limits, the data validation is conducted according to the data validation SOP QC Criteria indicated by check marks ($\sqrt{\ }$) in the "Review COC For" column of Table II. The SDG samples are validated using the Region 2 data validation SOP in conjunction with the latest version of the WinCADRE QC reports. The data validation flags (J,R,U) are applied on Form I's as well on the CADRE Lotus 1,2,3 or XLS Spreadsheet. Corrections, if needed, are then made on the Lotus or XLS spreadsheet to ensure that all results on Form I's carry the same data validation and concentration flags as are on the Lotus or XLS Spreadsheet.

7.4 COC "Not Evaluated"

Acceptance limits for the analytes not present/spiked in the PE sample are not provided on the PES Scoring Evaluation Report. Such analytes will be marked as "Not Evaluated" in the PES Evaluation Column. These analytes will be validated much the same way as the "Failed Analytes".

The failed analytes and the analytes not present/spiked in the PE sample require data validation according to the QC criteria specified in Table II, and are identified by the TOPO in the TDF for the Case/SDG.

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Table I

Passed PES - All Contaminants of Concern are within the limits (Action Low ≤ PES Result ≤ Action High)

QC Criteria	Review COC for
Holding Time & Preservation	√
Initial Calibration	
Initial Calibration Verification	
CRQL Standard	$\sqrt{}$
Blanks-Initial & Continuing	
Preparation Blank	
ICP Interference Check Sample	
Pre- Digestion/Distillation Matrix Spike	
Post Digestion Spike	
Laboratory Duplicate	
Field Duplicates Comparison	$\sqrt{}$
Lab Control Sample	
ICP Serial Dilution	
Field Blank Contamination	$\sqrt{}$
Percent Solids	$\sqrt{}$
Transcription/Computation Check	
Raw Data	
Total vs. Dissolved Concentrations Comparison	√

- The CSF (Complete SDG File) audit will be completed before the PES validation strategy is applied.
- Comparison of the Lotus or XLS Spreadsheet must be after the PES validation strategy is applied. The Contract
- Compliance can be checked after the PES validation strategy is applied.

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Table II Failed PES - Contaminants of Concern are not within the limits

(PES Result ≤ Action Low, PES Result ≥ Action High **OR** The Limits Not Established)

QC Criteria	Review COC for
Holding Time & Preservation	$\sqrt{}$
Initial Calibration	
Initial Calibration Verification	
CRQL Standard	√
Blanks-Initial & Continuing	
Preparation Blank	√
ICP Interference Check Sample	
Pre- Digestion/Distillation Matrix Spike	√
Post Digestion Spike	
Laboratory Duplicate	$\sqrt{}$
Field Duplicates Comparison	$\sqrt{}$
Lab Control Sample	$\sqrt{}$
ICP Serial Dilution	$\sqrt{}$
Field Blank Contamination	$\sqrt{}$
Percent Solids	$\sqrt{}$
Transcription/Computation Check	$\sqrt{}$
Raw Data	
Total vs. Dissolved Concentrations Comparison	√

- The CSF (Complete SDG File) audit will be completed before the PES validation strategy is applied.
- Comparison of the Lotus or XLS Spreadsheet must be after the PES validation strategy is applied.
- The Contract Compliance can be checked after the PES validation strategy is applied.

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8.0 **Sampling Trip Report**

The sampler prepares a Sampling Trip Report for each sampling event and sends it to the RSCC. The report provides details of all activities performed for each sampling event on the Superfund site. It also lists the field QC samples such as Field Duplicates, Field/Rinse Blanks, sampling time and date for each sample, and samples associated with each field/rinse blank. The validator must use this information to evaluate the Field Duplicate pairs as well as the samples associated with contaminated Field/Rinse Blanks.

9.0 Telephone Record Log (Appendix A.3)

A Telephone Record Log (Appendix A.3) must be written by the data validator when a deliverable is missing or a clarification is needed about a lab procedure. The data validator should <u>outline</u> a basic profile of the Case on the Telephone Record Log Form, clearly indicating the reason(s) for inquiry and forward this Form to CLP PO/TOPO who will contact the lab to receive the missing document or information. The original Telephone Record Log is kept in the data package and a copy attached to the Data Review Narrative.

10.0 Request for Re-Analysis (Appendix A.6)

Data validator must note all items of contract non-compliance in the Data Review Narrative. If holding times and sample storage times have not been exceeded, the Project Officer (PO) may request re-analysis if items of non-compliance are critical to data assessment. Requests are to be made on "CLP Re-Analysis Request/Approval Record" form (Appendix A.4).

11.0 CLP Data Assessment Summary Form (Appendix A.7)

Fill in the total number of analytes performed by different methods and the number of analytes rejected (R) or flagged (J) as estimated due to corresponding quality control criteria. Place an "X" in boxes wherever analyses were not performed, or criteria do not apply.

12.0 Data Review Log:

It is recommended that the data validator maintain a log of the reviews completed to document:

- a. Case number
- b. SDG # (s)
- c. number of samples
- d. matrix of samples
- e. contract laboratory
- f. site name
- g. start-date of the data case review
- h. completion-date of the data case review
- i. actual hours spent
- j. reviewer's signature

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13.0 Record of Communication -

This is a Regional document prepared and provided by the RSCC for each data package. The ROC indicates the Case #, site name, samples and sample matrix and the laboratory name. The presence of a ROC in a data package is an indication that the package has been reviewed by the RSCC for completeness and is ready for data validation.

14.0 **Forwarded Paperwork**

Upon completion of review, the following are to be forwarded to EPA for final review:

- a. Data package
- b. Completed data assessment checklist (Appendix A.1, original)
- c. Original and a copy of completed data review narrative Appendix A.2)
- d. CLASS Contract Compliance Screening (CCS) report
- e. Telephone Record Log (Appendix A.3)
- f. Field Duplicates Form (Appendix A.4)
- g. Total/Dissolved Concentrations Form (Appendix A.5)
- h. CLP Re-analysis Request/Approval Record Form (Appendix A.6)
- i. Data Assessment Summary Form (Appendix A.7)
- j. CADRE Spreadsheet on a computer diskette.

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ACRONYMS

AA Atomic Absorption

AOC Analytical Operations/Data Quality Center CADRE Computer-Aided Data Review and Evaluation

CCB Continuing Calibration Blank
 CCS Contract Compliance Screening
 CCV Continuing Calibration Verification
 CLP Contract Laboratory Program

CO Contracting Officer
COC Contaminants of Concern
CRI CRQL Check Standard

CRQL Contract Required Quantitation Limit

CSF Complete SDG File CVAA Cold Vapor AA

DART Data Assessment Rapid Transmittal

DAT Data Assessment Tool

DF Dilution Factor

DQO Data Quality ObjectiveICB Initial Calibration BlankICP Inductively Coupled Plasma

ICP-AES Inductively Coupled Plasma - Atomic Emission Spectroscopy

ICP-MS Inductively Coupled Plasma - Mass Spectrometry

ICS Interference Check Sample
 ICV Initial Calibration Verification
 LCS Laboratory Control Sample
 LRS Linear Range Sample
 MDL Method Detection Limit

NIST National Institute of Standards and Technology
OERR Office of Emergency and Remedial Response
OSWER Office of Solid Waste and Emergency Response

PB Preparation Blank
PE Performance Evaluation
%D Percent Difference
%R Percent Recovery

%RI Percent Relative Intensity

%RSD Percent Relative Standard Deviation

%S Percent Solids
PO Project Officer
QA Quality Assurance

OAPP Ouality Assurance Project Plan

QC Quality Control

RPD Relative Percent DifferenceRSCC Regional Sample Control Center

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SDGSample Delivery GroupSMOSample Management OfficeSOPStandard Operating Procedure

SOW Statement of Work
TAL Target Analyze List

TR/COC Traffic Report/Chain of Custody Documentation

Inorganic Target Analyze List And Contract Required Quantitation Limits (CRQLs)

Analyze CAS Number	ICP-AES CRQL Water Ug/L	ICP-AES CRQL Soil mg/kg	ICP-MS CRQL Water Ug/L
Aluminum 7429-90-5	200	20	
Antimony 7440-36-0	60	6	2
Arsenic 7440-38-2	10	1	1
Barium 7440-39-3	200	20	10
Beryllium 7440-41-7	5	0.5	1
Cadmium 7440-43-9	5	0.5	1
Calcium 7440-70-2	5000	500	
Chromium 7440-47-3	10	1	2
Cobalt 7440-48-4	50	5	1
Copper 7440-50-8	25	2.5	2
Iron 7439-89-6	100	10	
Lead 7439-92-1	10	1	1
Magnesium 7439-95-4	5000	500	
Manganese 7439-96-5	15	1.5	1
Mercury 7439-97-6	0.2	0.1	
Nickel 7440-02-0	40	4	1
Potassium 7440-09-7	5000	500	
Selenium 7782-49-2	35	3.5	5
Silver 7440-22-4	10	1	1
Sodium 7440-23-5	5000	500	
Thallium 7440-28-0	25	2.5	1
Vanadium 7440-62-2	50	5	1
Zinc 7440-66-6	60	6	2
Cyanide 57-12-5	10	2.5	

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Appendix A.1

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Site:
Case #:
SDG #:
Samples: Soil Water

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				YES NO N/A	
A.I.I	Contract Co	ompliance Screenin	g Report		
	Prese	ent?		[]	
	<u>ACTI</u>	ON: If no, contac	t RSCC/PO.		
A.I.2	Record of (Communication (fro	m RSCC)		
	Prese	ent?		[]	
	<u>ACTI</u>	ON: If no, reques	t from the RSCC.		
A.1.3	Sampling T	rip Report			
	Prese	ent and complete?		[]	
	<u>ACTI</u>	ON: If no, contac	t RSCC/PO.		
A.I.4	Chain of Cu	ustody/Sample Traff	<u>ic Report</u>		
	Prese	ent?		[]	
	Legib	ole?		[]	
	Signa prese	ature of sample custoent?	dian	[]	
	•	ON: If no, contact RS	SCC/WAM/PO.	·	
A.I.5	Cover Page	<u>.</u>			
Α.Ι.Ο	_				
	Prese	ent?		[]	
		Cover Page properly			
		he verbatim signed b ager or the manager's		[]	
	on th	ne sample identification e Cover Page agree ification numbers on:	with sample		
	(a) T	raffic Report Sheet?		[]	

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SOP:	HW-2	Revision 13	Appendix A.1	Sept. 2006
				YES NO N/A
	(b) For	m l's?		[]
		Is the number of samples on the Page the same as the number of samples on the Traffic Report s and the Regional Record of Cor (ROC) for the data Case?	of heet	[]
		ACTION: If no for any of the above, prepared the second Log and confor re-submittal of the corrected from the laboratory.	tact RSCC/PO	
A.1.6	SDG N	Narrative, DC-1 & DC-2 Form		
		Is the SDG Narrative present?		[]
		Is Sample Log-In Sheet(Form D present and complete?	OC-1)	[]
		Is Complete SDG Inventory She present and complete?	eet(Form DC-2)	[]
		ACTION: If no, write in the Contract-Probl Non-Compliance Section of t Narrative.	lems/ he Data Review	
A.1.7	<u>Form</u>	I to XV		
A.1.7.	1	Are all the Form I through Form labeled with:	XV	
		Laboratory Name?		[]
		Laboratory Code?		[]
		RAS/Non-RAS Case No.?		[]
		SDG No.?		[]

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				YES NO N/A	
		Contract No.?		[]	
A.1.7.2	2	ACTION: If no for any of the above Contract Problem/Non-Co of the "Data Review Narra PO for corrected Form(s) After comparing values or against the raw data, do a transcription errors exceed reported values on the Formatten Point Problem (Problem Point Problem Problem Problem Problem Problem Problem Problem Problem (Problem Problem (Problem Problem Proble	ompliance Section ative" and contact from the laboratory. In Forms I-IX any computation/ Id 10% of the		
	(a) al	I analytes analyzed by ICP	P-AES?	[_]	
	(b) al	l analytes analyzed by ICP	P-MS?	_ [] _	
	(c) M	ercury?		_ 🗀	
	(d) C	yanide?		_ [_] _	
	and c	ON: s, prepare Telephone Reco contact CLP PO/TOPO for from the laboratory.	•		
A.1.8	Data hard/	<u>Data</u> shall not be validated wi /electronic copies of the data for samples and QC	associated		
A.1.8.	1	Digestion/Distillation Log			
		stion Log for ICP-AES XII)present?		[]	
		stion Log for ICP-MS XII) present?		[]	
		stion Log for mercury n XII) present?		[]	
		lation Log for cyanide n XII) present?		[]	
	Are n	H values for metals and			

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					YES NO N/A
		reported for s sample?	each		[]
		cent solids ca for soils/sed			□
			es present on the logs/bench sheets	?	[]
			ust include weights, volunthe reported results.	mes,	
A.1.8.	2 Is real-tim	the analytica e printout	al instrument s present for:		
	ICP-AE	S?			[]
	ICP-MS	?			[]
	Mercury	/?			□
	Cyanide	e?			[]
	and inst	aboratory be trument raw o ary to suppor and QC ope	data printouts t all sample		
	Legible?				□
	Properly	labeled?			□
		eld samples, d QC sample	QC samples s present on:		
	Digestion	n/Distillation l	og?		[]
	Instrume	nt Printouts?			□

ACTION:

If no for any of the above questions in Section A.1.8.1 and Section A.1.8.2, write Telephone Record Log and contact TOPO/PO for re-submittal from the laboratory.

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					YES NO	<u>N/A</u>
(E	Examine s determine	ample Traffic R	eports and d	(Aqueous and soil samples) igestion/distillation logs to ample collection date to the sample		
A.1.9.	1 (Cyanide dis	stillation(1	4 days)exceeded?	_ 🗀 _	
	J	Mercury and	alysis(28 d	days) exceeded?	_ [] _	
	(Other Meta	ls analysis	s(180 days)exceeded?	[_]	
	nd flatif samp NOTE: In additional list of a which exist of a which exist on the number of the numb	reject (R) as ag as estimated as estimated as estimated as estimated as estimated as estimated as estimated. Report for ber of days that the sample prepares list to the data	the data, analytes alding times may each sample twere exceed plection date ation date).	ust e		
A.1.9.	2	ls pH of aqı	ueous san	nples for:		
	Metals	Analysis	<u><</u> 2?		[]	_
	Cyanio	de Analysis	<u>></u> 12?		□	
		DN: or any of the steets as "R"				
A.1.9.3	Is the c	cooler temp	erature <u><</u>	10 C°?	[_]	
		DN : er temperatu tects as "UJ'				
A.1.10) Final I	Data Corre	<u>ctness</u> - F	Form I		

A.1.10.1 Are Form I's for all samples

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				<u>YES</u>	<u>NO</u>	<u>N/A</u>	
	presen	t and complete?		[]			
	Log an	N: repare Telephone Record d contact CLP PO/TOPO for tal from the laboratory.					
A.1.10		/erify there are no calculation a eported on Form I's. Circle on	•				
].	s the calculation error less that	n 10% of the correct result?	[]			
		Are results on Form I's reported MG/KG for soils)?	d in correct units (ug/L for ac	queous a]	and]		<u> </u>
	A	Are results on Form I'S reporte	d by correct significant figu	ures?[_]		_
		Are soil sample results on Form corrected for percent solids?	n l's	[]] _		
		Are all "less than MDL" values by the CRQLs and coded with "	•	[]] _		
	b	Are values less than the CRQL out greater than or equal to the DLs flagged with "J"?		[]] _		
		Are appropriate contractual qua ontrol and Method qualifiers us	-	[] _		
	Ī p	ACTION: f no for any of the above questorepare Telephone Record Log CLP PO/TOPO for corrected date	g, and contact				
A.1.10	8 0	Do EPA sample identification nand the corresponding laborate sample identification numbers on the Cover Page, Form I's and the raw data?	ory match	[] _		

Was a brief physical description

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			YES NO N/A	7
	of the samples before an digestion given on the Fo		[]	_
	Was any sample result o mercury/cyanide calibrati or the ICP-AES/ICP-MS I diluted and noted on the	on range inear range	[]	
	ACTION: If no for any of the above the Contract-Problem/No Section of the Data Review	n-Compliance		
A.1.11 <u>Initia</u>	al Calibration			
A.1.11.1	Is a record of at least 2 p (A blank and a standard) present for ICP-AES ana	calibration		
	Is a record of at least 2 p (a blank and a standard)calib present for ICP-MS analy	ration	[]	_
	Is a record of at least 5 p (a blank & 4 standards)prese		[]	
	Is a record of at least 4 p (a blank & 4 standards)prese		[]	
	ACTION: If incomplete or no initial was performed, reject (R) the associated data (determine)) and red-line		
	Is one initial calibration st at the CRQL level for cya mercury?		[]	
	ACTION: If no, write in the Contract Non-Compliance Section Review Narrative.			
Δ1112	Is the curve correlation			

coefficient \geq 0.995 for:

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SOP: HW-2	Revision 13	Appendix A.1	Se	pt. 200)6
			YES	NO N	1/ <u>A</u>
	Mercury Analysis?		[]		
	Cyanide Analysis?		[]		
	ICP-AES(more than 2	point Calib.)?	[]		
	ICP-MS (more than 2	point calib.)?	г 1		
	ACTION: If no, qualify the a results > MDL as est non-detects as "UJ". NOTE: The correlation coefficient be calculated by the data vusing standard concentration corresponding instrument reabsorbance, peak area, peak	imated "J" and t shall validator ons and the esponse (e.g.			
A.1.12	Initial and Continuing	Calibration Verification	- Form II	<u>IA</u>	
A.1.12.1	Present and complete metal and cyanide?	e for every	[]		
	Present and complete and ICP-MS when both were used for the sa	these methods	[]		
	ACTION: If no for any of the Telephone Record Log for re-submittal fro	and contact PO/TOPO			
A.1.12.2	Was a Continuing Cal Verification perform 10 samples or every whichever is more fr	ned every 2 hours	[_] _	
	ACTION: If no for any of the in the Contract-Prob Section of the Data	olem/Non-Compliance			
A.1.12.3	Was an ICV or a mid- distilled and analyz of cyanide samples?	_	[]		

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			<u>YES</u>	NO N/A				
	Section of the Dat	the above, write coblem/Non-Compliance and MDL as estimated (J).						
A.1.12.2	Circle on each Form IIA that are outside the con-	•						
	Are ICV/CCVs within co	ntrol limits for:						
	Metals - 90-110%	R?	[]					
	Hg - 80-120%R?	•	[]					
	Cyanide - 85-115%	R?	[]					
	ACTION: If no, qualify all samples between a previous technically acceptable CCV standard and a subsequent technically acceptable CCV standard as follows as follows:							
	if the ICV/CCV %R is be Qualify only positive res between 111-125%(121 red-line only detects if the recovery is CN). Reject (R) and red	all detects and non-detects, etween 75-89%(65-79% for Hg; ults(≥ MDL) as "J" if the ICV/CC-135% for Hg;116-130% for CN greater than 125% (135% for He-line all associated results (hits as less than 75%(65% for Hg;70%)	V %R is). Reject (R) and Hg; 130% for and non-	,				
	NOTE: For ICV that does not fall within qualify all samples reported from							
A.1.12.3	Was the distilled ICV or standard for cyanide wit limits (85-115%)?	<u> </u>	[]					
	ACTION: If no, Qualify all cyanide	results ≥ MDL as "J".						

A.1.13 CRQL Standard Analysis - Form IIB

A.1.13.1 For each ICP-AES run, was a CRI

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			YES NO N/A	
	RQL or MDL when $MDL > CRC$	QL)		
star	ndard analyzed?		[]	
	(Note:CRI is not required Ca, Fe, Mg, Na and K.)	d for Al, Ba,		
	Ca, re, mg, Na and R.)			
	For each ICP-MS run, was	a CRI		
	(CRQL or MDL when MDL > CRO			
	analyzed for each mass/iso			
	for the analysis?	•	[]	
	·			
	For each mercury run, was	a CRQL		
	standard analyzed?		[]	
		0.7.01		
	For each cyanide run, was a	a CRQL		
	standard analyzed?		LJ	
	ACTION:			
	If no for any of the above, w	vrite		
	this deficiency in the Contra			
	Non-Compliance Section of			
	Narrative, inform CLP PO a	nd flag results		
	in the affected ranges (dete	cts <2xCRQL)as J		
	and non-detects UJ.	•		
	affected ranges are:	22.01		
	-AES Analysis - *True Value + (
	-MS Analysis - *True Value <u>+</u> (cury Analysis - *True Value <u>+</u> (
	inide Analysis - *True Value \pm (
Oya	* True value of the CRQL			
A.1.13.2	Was a CRQL standard ana	lyzed after the		
	ICV/ICB, before the final CO	CV/CCB and		
	once every 20 analytical sa	mples in		
	the analytical run for each a	nalysis?	[]	
	ACTION:			
	If no, write in the Contract F			
	Non-Compliance Section of	the		
	"Data Review Narrative".			
A.1.13.3	Circle on each Form IIB all	nercent		
٦.١.١٥.٥	recoveries that are outside	•		
		uic		
	acceptance windows.			

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SOP: HW-	2 Revision 13	Appendix A.1	Sej	ot. 2006	
	Is the CRQL standard within limits for:	n control	<u>YES</u>	NO N/A	
	Metals(ICP-AES/ICP-MS)-	70 - 130%?	[]		
	Mercury- 70 - 130%?		[]		
	Cyanide - 70 - 130%?		[]		
	ACTION: If no, flag detects <2xCRQL non-detects as "UJ" if the C recovery is between 50-69% detects <2xCRQL if the recovery and ≤180%. If the recovery is exclusive, reject(R) and red-line detects < 2xCRQL, and flag 2xCRQL and ICV/CCV. Rej detects <2xCRQL and flag but < ICV/CCV if the recovery	CRQL standard %. Flag(J) only covery is between covery is less than e non-detects and g (J) detects between ject and red-line only (J)detects <u>></u> 2xCRQL			
	NOTE: 1. Qualify all field samples a previous technically ac the CRQL standard and a s analysis of the CRQL standard. Flag (J) or reject (R) on sample results on Form I' raw data are within the a and the CRQL standard is acceptance windows. 3. The samples and the CRQL analyzed in the same analyzed.	sceptable analysis of subsequent acceptable adard aly the final s when Sample affected ranges outside the standard must be			
A.1.14 <u>Init</u> i	ial and Continuing Calibratio	on Blanks - Form III			
A.1.14.1	Present and complete for all the instruments used for the metals and cyanide analyse	e	[]		
	Was an initial Calibration Bl analyzed after ICV?	lank	[]		
	Was a continuing Calibratio analyzed after every CCV a 10 samples or every 2 hour is more frequent?	and every	[]		
	Were the ICB & CCB values reported on Form III and fla	_			

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			<u>YES</u>	NO N/A	
	using MDLs from direct a	nalysis(Preparation			
	Method "NP1")?	, ,	[]		
	(Check Form III agains	t the raw data)	- <u> </u>		
	ACTION:				
	If no, inform CLP PO/TO	PO and make a note			
	in the Contract-Problems				
	Section of the "Data Rev	•			
A.1.14.2	Circle with red pencil on	each Form III			
, <u>.</u>	all Calib. Blank values th				
	an Gano. Blank values in	at aro.			
	> /\	IDL but ≤ CRQL			
	<u>-</u>	.52 501 <u>5</u> 5.102			
	> C	CRQL			
A.1.14.2.1	When MDL < CRQL, is a	ıny Calib. Blank			
	value > MDL but < CRQL	•		[]	
	ACTION:				
	If yes, change sample re	culto > MDI			
		_			
	but < CRQL to the CRQL				
	Do not qualify non-detection	is.			
Λ 1 14 2 2 \W	hen MDL < CRQL, is any	Calib Blank			
	lue > CRQL?	Calib. Dialik		г 1	
va	ide > CRQL!			LJ	
	ACTION:				
		line the			
	If yes, reject (R) and red				
	associated sample result				
	but <icb blank="" ccb="" res<="" td=""><td></td><td></td><td></td><td></td></icb>				
	detects > ICB/CCB blank				
	< 10xICB/CCB value. Ch				
	results \geq MDL but \leq the (CRQL to CRQL			
	with a "U".				
	_				
	any Calibration Blank valu	ie			
be	elow the negative CRQL?			[]	
	ACTION:				
	If yes, flag (J) as estimate	ed all			
	associated sample result	s > CRQL but			
	<10xCRQL.				

NOTE:

^{1.} For ICB that does not meet the technical QC Criteria, apply the action to all samples

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	2. For apply previ	rted from the analytical run. CCBs that do not meet the technic the action to all samples analyze ous technically acceptable analys osequent technically acceptable a in the analytical run.,	ed between a sis of CCB and	YES	NO N/A
A.1.1	.5	Preparation Blank NOTE: The Preparation Bla is the same as the calib	nk for mercury		
A.1.1	5.1	Was one Preparatio with and analyzed			
		Each Sample Delive	ery Group (SDG)?	[]	
		Each batch of the digested/distilled	<u>-</u>	[]	
		Each matrix type?		[]	
		All instruments us and cyanide analys		[]	
		ACTION: If no for any of t as estimated (J) a positive data <10x Preparation Blank	ll the associated		
		NOTE: If only one blank was and than 20 samples, then the analyzed are not estimate additional samples must be additional samples.	e first 20 samples ed(J),but all		
A.1.1		Circle with red pen all Prep. Blank val	cil on each Form III ues that are:		
		> MDL but	\leq CRQL, and		
		> CRQL			
A.1.1	5.2.1	When MDL < CRQL, i value > MDL but <	s any preparation bland CRQL?	k	[]

<u>ACTION</u>:

If yes, change sample result \geq MDL

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			<u>YES</u>	NO	<u>N/A</u>
	but < CRQL to CRQL w	ith a "U".			
A.1.15.2.2	When the MDL < CRQL, Blank value greater			_ [.	1
	If yes, is the Prep. greater than the value Field Blank collected the SDG samples?	ue of the associated		-	[]
	If yes, is the lowest that analyte in the a less than 10 times the Blank value?	associated samples		-	[]
	sample results greated than the Prep.Blank of detects > Prep. Blank	nd red-line all associ er than the CRQL but l value. Flag as "J" k value but <10xPrep.B > MDL but < CRQL, rep	ess lank.		
	If the Prep. Blank value in the qualify the sample reprep. Blank criteria	esults due to the	same		
	NOTE: Convert soil sample result wet weight basis to compare Prep. Blank result on Form	e with the soil			
A.1.15.2.3	Is the Prep. Blank c below the negative C			[]
	ACTION: If yes, flag (J) all sample results less qualify non-detects a	than 10xCRQL.			
A.1.15.2.4	When the MDL is great CRQL, is the preparation on Forthan two times the MI	tion blank m III greater		_ [_]

<u>ACTION</u>:

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	If yes, reject (R) and red positive sample results wi raw data less than 10 time Preparation Blank value.	th sample	YES	<u>NO</u>	N/A
A.1.16	<pre>ICP-AES/ICP-MS Interferenc NOTE:Not required for CN, Hg, A</pre>		5)- For	m IV	
A.1.16.1	Present and complete?		[]		
	Was ICS analyzed at the beand end of each analytical once for every 20 analytic	run, and	[]		
	Was ICS analyzed at the be the ICP-MS analytical run?	ginning of	[]		
	ACTION: If no, flag as estimated (sample results.	J) all			
A.1.16.2	ICP-AES Method				
A.1.16.2.1	ICSA solution: For ICP-AES, are the ICSA values within the control of the true/established mean	limits <u>+</u> of CRQL	[]		
	If no for any of the above sample concentration of Al or Mg in the same units (u greater than or equal to i concentration in the ICSA Form IV?	, Ca, Fe, g/L or MG/KG) ts respective	[]		
	ACTION: If yes, apply the followin all samples analyzed betwe technically acceptable ana ICS and a subsequent techn analysis of the ICS in the	en a previous lysis of the ically acceptable			

Flag (J) as estimated only sample results \geq MDL

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			YES	<u>NO</u>	<u>N/A</u>	
	for which the ICSA "Found" val (True value+CRQL). Do not qual If the ICSA "Found" value is 1 (True value-CRQL), flag non-de detects as "J".	ify non-detects. ess than				
A.1.16.2.3	ICSAB Solution For ICP-AES, are all analyte r ICSAB within the control limit of the true/established mean v	s of 80-120	[]			
	If no for any of the above, is sample concentration of Al, Ca or Mg in the same units (ug/L greater than or equal to its r concentration in the ICSAB Sol Form IV?	, Fe, or MG/KG) espective	[]	_		
	ACTION: If yes, apply the following ac all samples analyzed between a technically acceptable analysi ICS and a subsequent technical analysis of the ICS in the ana	previous s of the ly acceptable				
	Flag (J) as estimated those as sample results ≥ MDL for which analyte recovery is greater th ≤ 150%. If the ICSAB recovery 50-79%, qualify sample results and non-detects as "UJ". Reject all sample results (detects & which the ICSAB analyte recove 50%. If the recovery is above and red-line only positive res	the ICSAB an 120% but falls within > MDL as "J" t (R) and red-line non-detects) for ry is less than 150%, reject (R)				
A.1.16.3	ICP-MS Method					
A.1.16.3.1	ICSA solution: For ICP-MS, are the ICSA "For values within the control li of the true/established mean vacation: If no, apply the following act samples reported from the analysis."	imits of <u>+</u> CRQL value? ion to all	[]			
	Flag (J) as estimated only samif the ICSA "Found" value is g (True value+CRQL). Do not qual If the ICSA "Found" value is 1 (True value-CRQL), flag the as detects as "J" and non-detects	reater than ify non-detects. ess than sociated sample				

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			<u>YES</u>	<u>NO</u>	<u>N/A</u>
A.1.16.3.3	ICSAB Solution For ICP-MS, are all analy in ICSAB within the contr 80-120% of the true/estak value, whichever is great	col limits of olished mean	[]		
	ACTION: If no, apply the followir samples reported from the				
	Flag (J) as estimated the sample results > MDL for analyte recovery is great < 150%. If the ICSAB reconstruction is sample results > MDL. Results and red-line only detects of the recovery is and red-line only detects.	which the ICSAB ter than 120% but overy falls within ated the associated ject (R) and red-line and non-detects for recovery is less than above 150%, reject (R)			
A.1.17	Spiked Sample Recovery: Property Service: Not required for Ca, Mg				
A.1.17.1	Was Matrix Spike analysis	s performed:			
	For each matrix type?		[]		
	For each SDG?		[]		
	On one of the SDG samples	3?	[]		
	For each concentration ratio (i.e., low, med., high)?	ange	[]		
	For each analytical Metho (ICP-AES,ICP-MS, Hg, CN)		[]		
	Was a spiked sample preparanalyzed with the SDG same		[]		
	ACTION: If no for any of the above estimated(J)all the posit for which a spiked sample analyzed.	tive data			

NOTE:

If more than one spiked sample were analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.

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			<u>YES</u>	NO	<u>N/A</u>	
A.1.17.2	Was a field blank or for the spiked sample			[]		
	ACTION: If yes, flag (J) as edata of the associate which field blank or for the spiked sample	d SDG samples for PE sample was used				
A.1.17.3	Circle on each Form V recoveries that are of control limits (75-12 sample concentrations times the added spike	outside the 5%) that have less than four				
	Are all recoveries will control limits when so concentrations are leequal to four times to concentrations? NOTE: Disregard the out of contractions for analytes who concentrations are greater equal to four times the specific control of the sequence of t	cample ess than or the spike rol spike cose contact than or	[]			
	Are results outside t (75-125%)flagged with on Form I's and Form ACTION: If no for any of the the Contract - Proble	Lab Qualifier "N" VA? above, write in ems/Non-Compliance	[]			
	Section of the Data R	Review Narrative.				
A.1.17.4	<u>Aqueous</u>					
	Are any spike recover	ies:				
	(a) less than 30%?			[]		
	(b) between 30-74%?			[]		
	(c) between 126-150%?			[]		
	(d) greater than 150%	?		[]		
	ACTION: If the matrix spike r 30%,reject (R) and re aqueous data (detects between 30-74%, quali aqueous data > MDL as	d-line all associated & non-detects). If fy all associated				

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SOP: HW-2	Revision 13	Appendix A.1	Se	pt. 200	6				
			YES	NO N	<u>/A</u>				
	as "UJ". If between 126-150 all data \geq MDL as "J". If greject (R) and red-line all	greater than 150%,	L.						
	(NOTE: Replace "N" with "J", "R" as appropriate.)								
A.1.17.5	Soil/Sediment								
	Are any spike recoveries:								
	(a) less than 10%?			[]					
	(b) between 10-74%?			[]					
	(c) between 126-200%?			[]					
	(d) greater than 200%?			[]					
	ACTION: If yes for any of the above as follows:	e, proceed							
	If the matrix spike recover than 10%,reject (R) and red associated data (detects & if between 10-74%,qualify a data \geq MDL as "J" and non-dif between 126-200%, flag (data \geq MDL as "J" If greate (R) and red-line all associated (NOTE:Replace "N" with "J" or	d-line all non-detects); all associated detects as "UJ"; J) all associated er than 200%, reject ated data > MDL.							
A.1.18	<u>Lab Duplicates</u>) - Form VI								
A.1.18.1	Was the lab duplicate analy	sis performed:							
	For each SDG?		[]						
	On one of the SDG samples?		[]						
	For each matrix type?		[]						
	For each concentration rang (low or med.)?	ge	[]						
	For each analytical Method (ICP-AES/ICP-MS, Hg, CN)Used?		[]						
	Was a lab duplicate prepare analyzed with the SDG sampl		[]						

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			<u>YES</u>	NO N/A	
	ACTION: If no for any of the estimated all the SDG (detects & non-detected analysis was all the specific duplicate analysis was set to see the second secon	G sample results ts) for which the lab			
	NOTE: If more than one lab dupl were analyzed for an SDG, the associated samples be worst lab duplicate analy	then qualify ased on the			
A.1.18.2	Was a Field Blank or for the Lab Duplicate	-		[]	
	ACTION: If yes, flag as estimm SDG sample results (I for which Field Blanused for duplicate a	hits & non-detects) nk or PE sample was			
A.1.18.3	Circle on each Form that are:	VI all values			
	RPD > 20%, or				
	Absolute Difference	> CRQL			
	Are all values within limits (RPD < 20% or difference < ±CRQL)?		[]		
	If no, are all result control limits flagge (Lab Qualifier) on For all Form I's?	ed with an "*"	[]		
	ACTION: If no, write in the or Non-Compliance Section Review Narrative.				
	NOTE: The laboratory is not recreport on Form VI the RPD both values are non-detection.) when			

A.1.18.4 **Aqueous**

A.1.18.4.1 When sample and duplicate values are both $\geq 5 \text{xCRQL}$ (substitute MDL for CRQL when MDL > CRQL),

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	is any RPD > 20% but < 100	18.2	<u>YES</u>	<u>NO</u> []	<u>N/A</u>	
	is any RPD > 100%?			[]		
A.1.18.4.2	ACTION: If the RPD is > 20% but < flag (J) as estimated the sample data > CRQL. If the > 100%, reject (R) and red associated sample data > C (NOTE:Replace "*" with "J" or " When the sample and/or dup	associated RPD is I-line the CRQL. R" as appropriate.)				
	<pre><5xCRQL (substitute MDL for is the absolute difference and duplicate values:</pre>					
	> <u>+</u> CRQL?			[_1	
	> <u>+</u> 2xCRQL?			[_]	
	ACTION: If the absolute difference flag as estimated all the sample results > MDL but < and non-detects as "UJ". I difference is > 2xCRQL, red-line all the associate and detects > MDL but < 5x NOTE: 1. Replace "*" with "J", "UJ" or calculate the absolute difference is > CRQL and the calculate the absolute difference is > CRQL and the MDL, and use this difference is > MDL but < 5x NOTE:	associated 5 5xCRQL as "J" 6f the absolute 7 ieject (R) and 8cd non-detects 8cRQL. 7 "R" as appropriate.) 8 other value is non-detect, 8 rence between the value > CR	.QL			
A.1.18.5	Soil/Sediment					
A.1.18.5.1	When sample and duplicate are both \geq 5xCRQL (substitute CRQL when MDL > CRQL),					
	is any RPD \geq 35% but < 120)%?		[_]	
	is any RPD > 120%?			[1	
	ACTION•					

If the RPD is \geq 35% and < 120%, flag (J) as estimated the associated sample

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		the RPD is \geq 120%, reject the associated sample	<u>YES</u>	<u>NO</u>	<u>N/A</u>	
A.1.18.5.2	<5xCRQL(substitu	and/or duplicate value te MDL for CRQL when MDL > CRQL), difference between sample				
	$> \pm 2 \times CRQL$?			[]		
	> <u>+</u> 4 x CRQL			[]		
	flag all the ass but < 5xCRQL as If the absolute	difference is > 2 x CRQL, sociated sample results > MDL "J" and non-detects as "UJ". difference is > 4xCRQL, reject all the associated non-detects DL but <5xCRQL.				
	1. Replace "*" with 2. If one value is calculate the ak	"J", "UJ" or "R" as appropriate.) >CRQL and the other value is non-detect solute difference between the value > C use this difference to qualify sample	RQL			
A.1.19	Field Duplicat	<u>es</u>				
	Aqueous Field D	<u>uplicates</u>				
A.1.19.1	Was an aqueous is collected and as (Check Sampling Tri	-	[]			

ACTION:

If yes, prepare a Form (Appendix A.4) for each aqueous Field Duplicate pair. Report the sample and Field Duplicate results on Appendix A.4 from their respective Form I's. Calculate and report RPD on Appendix A.4 when sample and its Field Duplicate values are both > 5xCRQL. Calculate and report the absolute difference on Appendix A.4 when at least one value (sample or duplicate) is <5xCRQL. Evaluate the aqueous Field Duplicate analysis in accordance with the

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					<u>YES</u>	<u>NO</u>	N/A	
		QC criteria	stated	in Sections A.1.19.2 and $$	A.1.19.3.			
A.1.1	-	2. Do not calc 3.Substitute M 4.If one value non-detect, between the this the cri	ulate RPD DL for CR is >CRQL calculate value > C teria to	from Form I's to Appendix A.4. when both values are non-dete QL when MDL > CRQL. and the other value is the absolute difference RQL and the MDL, and use qualify the results. on the Form (Appendix A.4) es that have:				
		RPD <u>></u> 20%	or					
		Difference >	> <u>+</u> CRQI	ı				
		_	_	plicate values are tute MDL for CRQL when				
		is any RPD 2	<u>></u> 20%?			[_]	
		is any RPD 2	<u>></u> 100%?			[_]	
		the associate results \geq CF	ed samp RQL. If only t	but < 100%, flag (J) only ple and its Field Duplicate the RPD is \geq 100%, reject the associated sample and sult \geq CRQL.	te t(R)			
A.1.1	9.3	<5xCRQL (sub	stitute Lute dif	l/or duplicate value(s) MDL for CRQL when MDL >CRQL ference between sample),			
		> <u>+</u> CRQL?				[_]	_
		> <u>+</u> 2 x CRQI	7.			[_]	_
				ference is > CRQL,				

If the absolute difference is > CRQL, flag detects > MDL but < 5xCRQL as "J" and non-detects as "UJ". If the difference is > 2xCRQL, reject (R) and red-line non-detects

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	and results \geq MDL but $<$ 5xCR and its Field Duplicate.	QL of the sample	YES	<u>NO</u>	<u>N/A</u>	
	Soil/Sediment Field Dupli	<u>lcates</u>				
A.1.19.4	Was a soil field duplicate collected and analyzed? (Check Sampling Trip Report)	pair	[]			
	ACTION: If yes, for each soil Field pair proceed as follows:	Duplicate				
	Prepare Appendix A.4 for eapair. Report on Appendix A. Field Duplicate results in respective Form I's. Calculsample and its duplicate vathan 5xCRQL. Calculate and absolute difference when at (sample or duplicate) is < 5 Field Duplicate analysis in QC Criteria stated in Section	4 all sample and its MG/KG from their ate and report RPD whe lues are both greater report the least one value accordance with the				
	NOTE: 1. Do not transfer "*" from Form 2. Do not calculate RPD when both 3. Substitute MDL for CRQL when MD 4. If one value is >CRQL and the of value is non-detect, calculate absolute difference between the value > CRQL and the MDL, and a the criteria to qualify the res	values are non-detects. L > CRQL. ther the upply				
A.1.19.5	Circle on each Appendix A.4 values that have:	all				
	RPD \geq 35%, or Difference > When sample and duplicate v are both \geq 5xCRQL (substitut CRQL when MDL > CRQL),	ralues				
	is any RPD > 35% but < 120%	?		[_]	
	is any RPD > 120%?			[_1	

ACTION:

If the RPD is \geq 35% but < 120%,

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	flag only the associat and its Field Duplicat CRQL as "J". If the reject (R) and red-lir and its Field Duplicat	te results RPD is <u>></u> 120%, ne only the sample	YES	NO N/A	
A.1.19.6	When the sample and/or <5xCRQL (substitute MDL is the absolute differ and Field Duplicate:	for CRQL when MDL $>$ CRQL),			
	> <u>+</u> 2 x CRQL?			[]	
	> <u>+</u> 4 x CRQL?			[]	
	Sample and its Field I but <5xCRQL as "J" and If the difference is red-line non-detects a	4xCRQL, reject(R) and			
A.1.20	Laboratory Control Sam	mple (LCS)- Form VII			
A.1.20.1	Was one LCS prepared a	and analyzed for:			
	Each SDG?		[]		
	Each matrix type?		[]		
	Each batch samples dig		[]		
	For each Method(ICP-ABused?	LS, ICP-MS, Hg, CN)	[]		
	Was an LCS prepared are the samples? ACTION: If no for any of the action of the action of the samples are th	above, prepare and contact omittal of the as estimated all	[]		
	NOTE: If only one LCS was analyze	ed for			

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SOP: HW-2	Revision 13 A	ppendix A.1	Se	ept. 2006	
	more than 20 samples, then the firs 20 samples analyzed are not flagged but all additional samples must be qualified (J).	t	E <u>S</u>	NO N/A	
A.1.20.2	Aqueous LCS				
	Circle on each Form VII the I recoveries outside control li				
	NOTE: 1.Use digested ICV as LCS for 2.Use distilled ICV as LCS f				
	Is any LCS recovery:				
	Less than 50%?	_		[]	
	Between 50% and 79%?	_		[]	
	Between 121% and 150%?	_		[]	
	Greater than 150%?	_		[]	
	ACTION: If the LCS recovery is less to reject (R) and red-line all assample data (detects & non-detects as "US as "J" all non-detects as "US recovery is between 121-150%, detects as "J". if the recover than 150%, reject (R) and red	essociated etects); for ag detects To if the LCS flag only ery is greater			
A.1.20.3	Solid LCS				
	If an analyte's MDL is equal greater than the true value of disregard the "Action" below analyte even though the LCS is control limits.	f LCS, for that			
	Is the LCS "Found" value greathan the Upper Control Limit reported on Form VII?	ter	<u> </u>	[]	

ACTION:

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	If yes, flag (J) all the as detects > MDL as estimated		<u>YES</u>	NO N/A	
	Is the LCS "Found" value lothan the Lower Control Limin reported on Form VII?			[]	
	ACTION: If yes, flag detects as "J" non-dectes as "UJ".	and			
A.1.21	ICP-AES/ICP-MS Serial Dil NOTE: Serial dilution analysis is when the initial concentration is greater than 50 x MDL.	required only			
A.1.21.1	Was a Serial Dilution analy performed:	rsis			
	For each SDG?		[]		
	On one of the SDG samples?		[]		
	For each matrix type?		[]		
	For each concentration rang (low or med.)?	re	[]		
	Was a Serial Dilution sample analyzed with the SDG sample		[]		
	ACTION: If no for any of the above, as estimated (J) detects > all the SDG samples for white ICP Serial Dilution Analysis not performed.	MDL of .ch the			
A.1.21.2	Was a Field Blank or PE sam for the Serial Dilution Ana			[]	
	<pre>ACTION: If yes, flag as estimated (MDL of all the SDG sample)</pre>				
Δ 1 21 3	Circle on Form WIII the Per	cent Differences			

(%D) between sample results and its dilution results that are outside the control limits \pm 10%

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SOP: HW-2	Revision 13	Appendix A.1	Se	ept. 2006
	when initial concentr	ations <u>></u> 50 x MDLs.	<u>YES</u>	NO N/A
	Are results outside t limits flagged with a on Form VIII and all	n "E"(Lab Qualifier)	[]	
	ACTION: If no, write in the C Non-Compliance Sectio Review Narrative.			
A.1.21.4	Are any %D values:			
	> 10%?			[]
	≥ 100%?			[]
	if the %D is \geq 100%,			
	(NOTE: Replace "E" with	"J" or "R" as appropriate.)		
A.1.22	Total/Dissolved or In	organic/Total Analytes		
A.1.22.1	Were any analyses per dissolved as well as on the same sample(s) Were any analyses per inorganic as well as on the same sample(s)	total analytes ? formed for total analytes	_	[]
		ences between ic)and total s. Compute each x A.5 as a percent only when both of		
A.1.22.2	(2) greater than or e	qual to 5xMDL.		
n,1,22,2	concentration greater	than its		[]

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SOP: HW-2	Revision 13	Appendix A.l	Se	ept. 2006
			<u>YES</u>	NO N/A
A.1.22.3	Is any dissolved(or inorgation concentration greater than total concentration by mor	n its		[]
	ACTION: If the percent difference than 20%, flag (J) both di and total concentrations a the difference is more that and red-line both the value.	ssolved/inorganic as estimated. If an 50%, reject (R)		
A.1.23	Field Blank - Form I NOTE: Designate "Field Blank	u as such on Form I		
A.1.23.1	Was a Field/Rinsate Bank of and analyzed with the SDG		[]	
	If yes, is any Field/Rinsa absolute value of an analy greater than its CRQL(or 2	rte on Form I		[]
	If yes, circle the Field E on Form I that is greater CRQL, (or 2 x MDL when MDL > CRO	than the		
	Is any Field Blank value g than CRQL also greater tha Preparation Blank value?			[]
	If yes, is the Field Blank (> CRQL and > the prep. bl already rejected due to ot criteria?	ank value)	[]	

<u> ACTION</u>:

If the Field Blank value was not rejected, reject all associated sample data (except the Field Blank results) greater than the CRQL but less than the Field Blank value. Reject on Form I's the soil sample results whose raw values in ug/L in the instrument printout are greater than the CRQL but less than the Field Blank value in ug/L. Flag as "J" detects between the Field Blank value and 10xField Blank value. If the sample result > MDL but < CRQL, replace it with CRQL-U.

If the Field Blank value is less than the

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SOP: HW-2	Revision 13	Appendix A.1	Se	ept. 2	006	
	Prep.Blank value, do not results due to the Field		YES	<u>NO</u>	<u>N/A</u>	
	NOTE: 1. Field Blank result previous due to other criteria cannot qualify field samples. 2. Do not use Rinsate Blank a soils to qualify water same	ot be used to ssociated with				
A.1.24	Verification of Instrume	ental Parameters - Form	IX, XA,	XB, XI		
A.1.24.1	Is verification report p	present for:				
	Method Detection Limits	(Form IX-Annually)?	[]			
	ICP-AES Interelement Cor (Form XA & XB -Quarterly		[]			
	<pre>ICP-AES & ICP-MS Linear (Form XI-Quarterly)?</pre>	Ranges	[]			
	ACTION: If no, contact CLP PO/TO submittal from the labor					
A.1.24.2	Method Detection Limits -	- Form IX				
A.1.24.2.1	Are MDLs present on Form	n IX for:				
	All the analytes?		[]			
	All the instruments used	1?	[]			
	Digested and undigested samples and Calib.Blanks	5?	[]			
	ICP-AES and ICP-MS when instruments are used for same analyte?		[]			
	ACTION:					

If no for any of the above, prepare Telephone Record Log and contact CLP PO/TOPO for submittal of the MDLs from the laboratory. Report to CLP PO and write in the Contract Problems/ Non-Compliance Section of the Data Review Narrative if the MDL concentration is not less than ½ CRQL.

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SOP: HW-2	Revision 13	Appendix A.1	Sept. 2006	
A.1.24.2.2	Is MDL greater than tor any analyte?	the CRQL	<u>YES NO N/A</u> []	
	If yes, is the analyte on Form I greater tha the sample analyzed of whose MDL exceeds CRQ	an 5 x MDL for on the instrument	[]	
	ACTION: If no, flag as estimate values less than five the analyte whose MDI	e times MDL for		
A.1.24.3	Linear Ranges - Form	ХI		
A.1.24.3.1	Was any sample result the high linear range or ICP-MS?	_	[]	
	Was any sample result the highest calibrate for mercury or cyanic	ion standard	[]	
	If yes for any of the the sample diluted to result reported on Fo	o obtain the	[]	
	ACTION: If no, flag (J) as es affected detects (> Non Form I.			
A.1.25	ICP-MS Tune Analysis	- Form XIV		
A.1.25.1	Was the ICP-MS instrutuned prior to calibration		[]	
	ACTION: If no, reject (R) and sample data for which performed.			
A.1.25.2	Was the tuning solution scanned at least for consecutively?		[]	
	Were all the required spanning the analytic present in the tuning	cal range	[]	
	Was the mass resoluti	ion within		

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SOP: HW-2	Revision 13 Apper	ndix A.1		Sept	. 2006
			<u>YES</u>	<u>NO</u>	<u>N/A</u>
0.1 amu fo	r each isotope in the tuning solution?		[]		
	Was %RSD less than 5% for each isotope of each analyte in the tuning solution?		[]		
	ACTION: If no for any of the above, of all results > MDL associated Tune as estimated "J", and all associated with that Tune as	with that Il non-detects			
A.1.26	ICP-MS Internal Standards - 1	Form XV			
A.1.26.1	Were the Internal Standards at to all the samples and all QC samples and calibration stand (except the Tuning Solution)	C dards	[]		
	Were all the target analyte masses bracketed by the masse of the five internal standard		[]		
	ACTION: If none of the Internal Standarded to the samples, reject red-line all the associated a (detects & non-detects). If a standards were used but did a the analyte masses, reject (Nonly the analyte results not the internal standard masses	(R) and sample data internal not cover all R) and red-line bracketed by			
A.1.26.2	Was the intensity of an Interstandard in each sample with of the intensity of the same Standard in the calibration by	in 60-125% Internal	[]		
	If no, was the original sample two fold, Internal Standard a sample re-analyzed?		[]		
	Was the %RI for the two fold within the acceptance limits	-	[]		
	ACTION: If no for any of the above, as "J" and non-detects "UJ" analytes with atomic masses	of all the			
	atomic mass of the internal s	standard lighter			

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SOP: F	₩-2	Revision 13	Appendix A.2	Sept	2006
than	the a	atomic mass	ernal standard, and the s of the internal standard h Efected internal standard.	neavier	
A.1.2	7	Percent So	lids of Sediments		
A.1.2	7.1	Are percent	solids in sediment(s):		
		< 50%?			[]
		non-detects less than !	alify as estimated (J) all desorated of a sample that has perce 50%(i.e., moisture content great	ent solids	
			the sample results previously flagged OC criteria		
<u>Inor</u> Case#	-	c Data Re	view Narrative	Matrix: Soil	
SDG#			Lab:	Water	:
Sampl	ing T	eam:	Reviewer:	Other	
A.2.1	The	considered	Flags: flags may have been applied by the data user. This flag indicates the resu		
			_	_	
	R and	ked-Line -	A red-line drawn through a The red-lined data are know documented information and	n to contain signif	icant errors based on
	U -		This data validation qualif > MDL when associated blank		ample results
	Fully	Usable Data	- The results that do no usable.	ot carry "J" or "red	d-line" are fully

A.2.2 <u>Laboratory Qualifiers</u>:

The CLP laboratory applies a contractual qualifier on all

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Form I'S and the QC Form when a QC analysis is outside the control limits. These

Appendix A.2

SOP: HW-2 Revision 13

qualifiers are not applied on the Lotus or XLS spreadsheets. These qualifiers and their meanings are as follows:

- N: This qualifier indicates the lack of accuracy in the reported result, and is applied when matrix spiked sample recovery is outside the control limits.
- E: This qualifier indicates the the presence of interference, and is applied when the ICP serial dilution is outside the control limits.
- *: This qualifier indicate the lack of precision , and is pplied on Fom I'S and Form VI when the Lab Duplicate analysis is outside the control limits.
- **U:** This is a concentration qualifier that laboratory applies to a non-detected result which is essentially less than the Method Detection Limit(MDL). A non-detected result of an analyte is indicated by the Contract Required Quantitation Limit (CRQL) of that analyte suffixed with "U".
- ${\tt J:}$ This is also a concentration qualifier that laboratory applies to a positive result below the CRQL.

NOTE: The laboratory qualifiers are crossed out and replaced with the appropriate data validation qualifiers (J, R or U) by the data validator.

A.2.3.1	<u>Data Case Description</u> :
A.2.3.2	<pre>CSF Audit:</pre>

Standard Operating Procedure

USEPA Region 2

SOP: HW-2	Revision 13	Appendix A.2	Sept. 2006	
A.2.3.3	Technical Review:			
_				
A.2.3.4	Contract-Problem	Non-Compliance:		
				
-				
_				
-				

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SOP: HW-2 Revision 13 Appendix A.2 Sept. 2006 HWSS Reviewer: _____ Date:____ Signature Contractor Reviewer: Date: Signature Verified by: Date:_____ Signature CASE # Contract Laboratory Program REGION II/LABORATORY COMMUNICATION SYSTEM SDG # Telephone Record Log Date of Call: ESAT Reviewer/Date: _____ Type of Analysis: Inorganic Laboratory Name: Lab Contact: Call Initiated By: ___Laboratory _X_Region II Inquiry made in reference to data for the following sample number(s): Summary of Questions/Issues Discussed:

Summary of Resolution:

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	_	
Signature		Date:

FIELD DUPLICATES

Sample No. Field Duplicate No. Sample Matrix:

Lab Code: Case No.: SDG No.:

% Solids Sample: % Solids Duplicate:

Concentration Units (ug/l or mg/kg dry weight):

	Action Limit	Sample Concentration	С	Duplicate Concentration	C	RPD	Difference	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									

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Appendix A.1 Revision 13 SOP: HW-2 Sept. 2006 YES <u>NO</u> N/A Mercury Nickel Potassium Selenium Silver Sodium **Thallium** Vanadium Zinc Cyanide

Total/Dissolved Concentrations

Lab Code Case No. SDG No. Sample Matrix: Water

Concentration: ug/L

ANALYTE	TOTAL	С	DISSOLVED	С	DIFFERENCE	Q	м
ALUMINUM							
ARSENIC							
BARIUM							
BERYLLIUM							
CADMIUM							
CALCIUM							
CHROMIUM							
COBALT							
COPPER							
IRON							
LEAD							
MAGNESIUM							
MAGNESE							
MERCURY							
NICKEL							

POTASSIUM				
SELENIUM				
SILVER				
SODIUM				
THALLIUM				
VANADIUM				
ZINC				
CYANIDE				

CONTRACT LABORATORY PROGRAM CLP RAS RE-ANALYSIS REQUEST/APPROVAL RECORD

SECTION A (TO BE COMPLETED BY REGIONAL SENDING OFFICIAL)

Init:	iated By: Name, Affiliation, Phone Number	Case Number: ! OLM ! OLC											
Detail	s of Re-Analysis Request:	! ILM											
!	Laboratory Name /Contract Number:												
!	Affected Sample Number(s) and Fraction(s):												
!	Reason for Re-Analysis:												
!	Contract Statement of Work Citation*:												
!	Comments:												
!	* PROVIDE SOW CITATION THAT SUPPORTS THIS REQUEST												
	RE-ANALYSIS Billable () Not Billable	()											
!	Approved By:	Date:											
	Authorized Regional Sending CLP PO Signature												
SECT	TON B (TO BE COMPLETED BY SMO)												
Name	of SMO Contact Da	ate:											

Date of Labo	oratory Noti	fication	ı (Verbal):							_				
Re-analysis S	Start Date: _						Data Du	e Date: _						
			Sam	ple Ma	nagement O			ompleted	d form to:					
Distribtion: Final 9/3/99	(1) CLP	PO Cop	oy (2) Reg	ional Se	ending Offici	al Copy	(3) SMO I	File Copy	y (4) Labora	tory Copy				
Type of R	.eview:								ARY FORI	-		5)		
Site:						Name:					-			
Reviewer'					Nur	mber of	Sample	es:			ew Criter	ria		
	Holding Time	CRQL Std	Blanks	ICS	Spike Recovery	Dup.	Dup. Field	LCS	ICP Serial Dilution	% Solids	Internal Std. ICP-MS	Tuning ICP-MS	Total Analytes	Rejection
ICP-AES														

	Time	std		Recovery	Lab.	Field	Serial Dilution	Solids	Std. ICP-MS	ICP-MS	Analytes	%
ICP-AES												
ICP-MS												
Mercury												
Cyanide												
Total												

Analytes Flagged (J) as Estimated Due to Exceeding Review Criteria

	Holding Time	CRQL Std	Blanks	ICS	Spike Recovery	Dup. Lab.	Dup. Field	LCS	ICP Serial Dilution	% Solids	Internal Std. ICP-MS	_	Total Analytes	Rejection %
ICP-AES														
ICP-MS														
Mercury														
Cyanide														
Total		·												